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Issued May 21, 1909.

U. S. DEPARTMENT OF AGRICULTURE.

OFFICE OF PUBLIC ROADS—BULLETIN No. 35.

LOGAN WALLER PAGE, DIRECTOR.

THE PRESERVATION OF IRON AND STEEL.

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BY

ALLERTON S. CUSHMAN,
ASSISTANT DIRECTOR, OFFICE OF PUBLIC ROADS.

WASHINGTON:
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1909.

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LETTER OF TRANSMITTAL.

U. S. Department of Agriculture,
Office of Public Roads,
Washington, D. C., March 12, 1909.

Sir: I have the honor to transmit herewith a manuscript containing data which furnish a continuation of the investigations already published by this Office in Bulletin No. 30, on the corrosion of iron. The work done by the writer of the bulletin along this line of investigation has resulted in an entire change of the metallurgical method of procedure of a number of steel-rolling mills in this country. It is now possible to obtain an extremely pure iron for the manufacture of road culverts. This pure material is giving satisfaction to the manufacturers of culverts as well as to road builders for securing proper drainage of roadways.

In this manuscript, work is also described which has a direct bearing upon the preservation of fence wire, as well as upon the various methods of providing preservative coatings for different forms of iron and steel in general use. I consider this work to be of great value, and it is therefore requested that this manuscript be printed as Bulletin No. 35 of this Office.

Respectfully,

L. W. Page, *Director.*

Hon. James Wilson,
Secretary of Agriculture.

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THE PRESERVATION OF IRON AND STEEL.

INTRODUCTION.

It is significant of the present age that the tendency of thought among all nations is toward conservation of natural resources. Increasing consumption of the world's supplies and constant decay of materials menace the future of the human race. It is evident that carbon, from which we derive energy, and iron, which provides the principal means of applying energy, are the materials which should particularly engage the attention of those who are studying the problems of conservation. The annual production of pig iron in the United States alone grew from 14,009,870 tons in 1900 to 26,193,863 tons in 1907, and while for the year 1908, owing to industrial conditions, it decreased to about 16,000,000 tons, it seems sure to again show an increase in the near future. How much of this enormous and constantly increasing world's production of iron and steel is wasted for lack of adequate preservation? Where will the growth of demand stop, and how many years will the world's ore supply stand the drain upon it? These are questions of vast importance, the answers to which can only be vaguely guessed. One thing seems certain, namely, that civilization must learn to conserve more efficiently its stores of iron and steel already manufactured, and seek methods to prevent the almost resistless tendency of iron to return to its lethargic union with oxygen, from which it was won only by the consumption of vast quantities of the ever dwindling coal supply. It is not generally realized that about four tons of coal or its equivalent is used in preparing one ton of finished steel from the ore. The world's store of gold is not subject to loss by corrosion, but suffers to some extent from attrition, owing to the softness of the metal. If steel could by any means whatsoever be ennobled and thus protected from the inevitable decay due to corrosion, future conditions for all possible years to come could be viewed with complacency. It is said [a] that there exists at Delhi, India, an iron monument that, since the dim beginning of history, has been exposed to the weather without rust or decay, and yet this column has been provided with no protective coating, other than that which the atmosphere has itself formed upon it. Could we to-day, with all our boasted knowledge and our great pneumatic processes, build its like? It is probable that we could not, and yet an art is not necessarily lost forever. It is simply a question whether or not it is worth our while to rediscover it.

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[a] Wood, M. P., Rustless Coatings, 1905, p. 170.

THE CORROSION OF IRON.

It is the object of this paper to examine into the controlling factors that influence the causes and the cure of the corrosion of iron, and to inquire into the possible practical bearing of the results of recent research.

The writer has in previous papers[a] referred to the fact that the problem of the preservation of iron has three distinct phases. The first phase has to do with the manufacture of a metal highly resistant to corrosion, such as is represented by the iron column of Delhi or the splendid examples of hand-forged metal that have come down to us from past centuries, and with which we may contrast the pitted, lacelike condition of specimens of modern steel after only a few years' exposure to the atmosphere. The second phase concerns the general subject of protective coatings, which may consist of other metals, such as zinc, tin, copper, and lead, or of oil paints, varnishes, lacquers, and bituminous materials, or, finally, the production of a higher oxide on the surface, as in the Bower-Barf, Wells, and Speller processes. The third phase includes the interesting study of the passive condition which iron is capable of assuming, and the possibility of maintaining the surface in such an ennobled condition.

Many discussions[b] have arisen in regard to the several rival theories of corrosion, but the evidence which has been collected in numerous recent researches appears to the writer to prove beyond all argument that the corrosion of iron, like that of other metals, is an electrochemical phenomenon.

In considering the corrosion of iron it is important to remember that iron is a metal which readily combines with or dissolves nearly all the other elements. With possibly one or two exceptions, there are no elements that do not either dissolve in or combine readily with iron. It is also unique in the fact that very small quantities of impurities suffice to change its physical characteristics entirely. On account of this fact metallurgists scrutinize the hundredth of a per cent of some of the principal impurities that are generally associated with this metal. This is particularly true, for instance, of the element phosphorus. It is so important to modern metallurgy that the amount of phosphorus should be controlled in certain forms of steel that an animated discussion is going on at the present time between certain interests as to the control of the amount of phosphorus that steel shall carry, and the question at issue amounts to no more than a few hundredths of 1 per cent.

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[a] U. S. Dept. Agr., Office of Public Roads, Bul. 30, 1907; Trans. Am. Soc. for Testing Materials, 1907, **7**, 209; Trans. Am. Electrochem. Soc. 1907, **12**, 403.

[b] Am. Soc. Mech. Eng., Trans. 1894, **15**, 998; ibid., 1895, **16**, 350, 663; Zts. Elektrochemie, 1903, **9**, 442; Treadwell and Hall, Analytical Chemistry, 1907, p. 92; Manchester Lit. Phil. Mem., 1871, **5**, 104; Jour. Iron and Steel Inst., 1888, 129–131; Proc. Chem. Soc. (Lond.), 1906, **22**, 101; Jour. Chem. Soc. (Lond.), 1905, **87**, pt. 2, 1548; Jour. Am. Chem. Soc., 1903, **25**, 394; The Analyst, 1905, **30**, 232; Trans. Chem. Soc. (Lond.), 1906, **98**, 1356.

Manganese is also an element which is nearly always associated in modern metallurgy with iron and steel. Manganese decreases the electrical conductivity of iron, and as the percentage of manganese, starting from zero, rises, the electrical resistance increases up to a certain specific maximum. It will be seen that if the presence of manganese in iron raises the electrical resistance, any variation in the distribution of the manganese means that there will not be a constant electrical conductivity throughout its mass, or on any given surface. There is abundant evidence to show that manganese associates itself to a considerable extent with sulphur when both these impurities are present in steel.[a] That manganese sulphide shows a difference of electrical potential against iron is also well known. One who is familiar with the methods of modern metallurgy knows that the manganese is added for certain specific purposes, not as a rule quantitatively, but in accordance with the views of the iron master who has control of the mill or furnace. Moreover, the manganese is usually added by throwing lumps of ferromanganese into the molten metal, either in the furnace itself or in the ladle into which it has been poured. Chemists know the extreme care that has to be taken in order to get uniform mixtures of substances in the course of chemical operations. On the large scale on which metallurgical processes are conducted, even if it were possible to take great care in the mixing, it still happens that when iron is cooled from the molten state segregation takes place—that is to say, the impurities, although they may have been thoroughly mixed in the molten mass, do not remain homogeneously distributed after the metal is cooled.

For these reasons we must remember that in studying iron and steel from the standpoint of their stability, under the conditions of service, we are not dealing with homogeneous pure metal. It is not difficult for an electro-chemist to believe that when such material is immersed in or even brought into contact with an electrolyte, electrolysis will take place upon the surface, and thereby induce rapid corrosion. It is probable that the corrosion of all metals is more or less due to electro-chemical action. Before metals can be attacked at ordinary temperatures in the presence of water they must first pass into solution, and in passing into solution become ionized. This is true of copper, zinc, lead, and the other metals that suffer corrosion.

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[a] Trans. Am. Soc. for Testing Materials, 1908, 8, 74.

In accordance with the conceptions of Nernst[a] and the modern theory of solutions, all metals have a certain solution pressure which will operate until counterbalanced by the osmotic pressure. Iron, however, appears to differ from the other metals in one important respect. The corrosion of iron does not take place evenly and uniformly over the surface. On the contrary, it is a matter of common observation that iron corrodes rapidly at certain weak points, the effect produced being known as "pitting." That this effect is produced by local electrolysis would hardly be doubted, even if it were not possible to demonstrate it by the use of the ferroxyl indicator.

Early in his investigations the writer[b] observed that whenever a specimen of iron or steel is immersed in water or a dilute neutral solution of an electrolyte to which a sufficient quantity of phenolphthalein indicator has been added, a pink color is developed. If the solution is allowed to stand perfectly quiet, it will be noticed that the pink color is confined to certain spots or nodes on the surface. The pink color of the indicator is a proof of the presence of hydroxyl ions, and thus indicates the negative poles. It may be added that some specimens of steel exhibit this phenomenon much more quickly and distinctly than others.

Since phenolphthalein shows only the nodes where solution of iron and subsequent oxidation does not take place, Walker[c] suggested the addition of a trace of potassium ferricyanide to the reacting solution, in order to furnish an indicator for the ferrous ions whose appearance marks the positive poles. If iron goes into solution, ferrous ions must appear, which, with ferricyanide, form the well-known Turnbull's blue compound. If the reagent is stiffened with gelatin or agar-agar, diffusion is prevented and the effects produced are preserved. For this combined reagent, which indicates at one and the same time the appearance of hydroxyl and ferrous ions at opposite poles, the writer has suggested for the sake of brevity the name "ferroxyl." If the reagent has been properly prepared, the color effects are strong and beautiful. In the course of a few days the maximum degree of beauty in the colors is obtained, after which a gradual deterioration sets in.

In the pink zones, as would naturally be expected, the iron remains quite bright as long as the pink color persists. In the blue zones the iron passes into solution and continually oxidizes, with a resulting formation of rust. Even the purest iron develops the nodes in the ferroxyl indicator, but impure and badly segregated metal develops the colors with greater rapidity and with bolder outlines. This result would of course be expected, as in pure iron the formation of poles would be conditioned by a much more delicate equilibrium than in impure iron, where variations in concentration of the dissolved impurities would stimulate the electrolytic effects.

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[a] Jones, H. C., Elements of Physical Chemistry, 1907, p. 449.

[b] U. S. Dept. Agr., Office of Public Roads, Bul. 30, 1907.

[c] Jour. Am. Chem. Soc., 1907, 29, 1257.

The ferroxyl mounts are prepared as follows: A 1½ per cent solution of agar-agar is first made by dissolving a weighed quantity of powered agar in the requisite amount of water. This solution is boiled for one hour, fresh water being added to replace that lost by evaporation. It is then filtered while hot and 2 c. c. of standard phenolphthalein indicator added to every 100 c. c. solution, after which it is brought to a perfectly neutral condition by titration with a tenth-normal solution of potassium hydroxide (KOH) or hydrochloric acid (HCl), as the case may be. The addition of 7 c. c. of a 1 per cent solution of potassium ferricyanide to every 100 c. c. of solution is then made, and the ferroxyl reagent, while still hot, is ready to use. Enough of the reagent should be poured into a Petri dish to just cover the bottom, and the dish floated in cold water until the agar has jellied. A clean sample of iron is then placed on this bed of jelly and covered with the hot solution. After the final addition of agar the dish should not be moved until thoroughly cool. While the colors sometimes show up immediately, they usually require from twelve to twenty-four hours to attain their most perfect development. The mounts may be preserved for many months by keeping the surface of the agar covered with alcohol.

In the writer's opinion, these effects which are produced in the ferroxyl indicator constitute a visible demonstration of electrolytic action taking place on the surface of iron and causing rapid corrosion at the positive nodes. This electrolytic action, which is shown to take place whenever iron rusts, should not, however, be confused with the rapid destruction of steel in the neighborhood of escaped currents from high potential circuits used for electric lighting or tramways. In order to avoid such confusion of terms and ideas, it has been proposed to speak of the underlying electro-chemical cause of corrosion as autogenous[a] electrolysis or, more briefly, autoelectrolysis.[b] It is not the author's intention to collect at this time all the evidence which tends to prove that autoelectrolysis always takes place when iron rusts; this has already been done in numerous papers by several independent investigators.[c] The case may be allowed to rest on the evidence of the ferroxyl test, which at least is conclusive as to the fact that iron is going into solution and oxidizing more rapidly at certain points than at others.

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[a] Proposed by G. W. Thompson.

[b] Proposed by R. H. Gaines, Eng. News, 1908, **59**, 583.

[c] Whitney, Jour. Am. Chem. Soc., 1903, **25**, 394; Cushman, U. S. Dept. Agr., Office Public Roads, Bul. 30, 1907; Walker, Jour. Am. Chem. Soc., 1907, **29**, 1251.

The electro-chemical explanation of the corrosion of iron is not complicated, and so far has been found in accordance with all the facts. Briefly stated, the explanation is as follows: Iron has a certain solution tension, even when the iron is chemically pure and the solvent pure water. The solution tension is modified by impurities or additional substances contained in the metal and in the solvent. The effect of the slightest segregation in the metal, or even unequal stresses and strains in the surface, will throw the surface out of equilibrium, and the solution tension will be greater at some points than at others. The points or nodes of maximum solution pressure will be electro-positive to those of minimum pressure, and a current will flow, provided the surface points are in contact through a conducting film. If the film is water, or is in any way moist, the higher its conductivity the faster iron will pass into solution in the electro-positive areas, and the faster corrosion proceeds. Positive hydrogen ions migrate to the negative areas, negative hydroxyls to the positives. By a hydrogen ion is meant a dissociated hydrogen atom carrying its equivalent static electrical charge, which may be represented by the symbol $\overset{+}{H}$. The hydroxyl ion is written $\overset{-}{OH}$. Water, which may be expressed by the symbol HOH, is made up of the dissociation products $\overset{+}{H}$ and $\overset{-}{OH}$. An acid like hydrochloric acid (HCl) dissociates into $\overset{+}{H}$ and $\overset{-}{Cl}$. An acid is always highly dissociated in solution, while water itself is only slightly dissociated. This explains why the presence of an acid increases the concentration of the hydrogen ions. Ionization always takes place in every solution of an inorganic compound, and even the purest water is slightly dissociated into its constituent ions $\overset{+}{H}$ and $\overset{-}{OH}$. The more ionized a solution is, the higher its electrical conductivity, and the more rapid the damage to the underlying iron.

If the concentration of the hydrogen ions is sufficiently high, which, as has been shown, is only the same as saying if the solution is sufficiently acid, the hydrogen ions will exchange their electrostatic charges with the iron atoms sweeping into solution, and gaseous hydrogen is seen escaping from the system. This takes place whenever iron is dissolved in an acid. If, however, as is usual in ordinary rusting, the acidity is not high enough to produce this result, the hydrogen ions will polarize to a great extent around the positive nodes without accomplishing a complete exchange, and the so-called electrical double layer of Helmholtz will be formed.[a] This polarization effect resists and slows down the action. Nevertheless, although it can not be seen, some exchange takes place and iron slowly pushes through, as is shown by the development of the blue nodes in the ferroxyl test. For every exchange of static charge between iron and hydrogen at the positive node, a corresponding negative hydroxyl ion appears at the negative node which is shown in pink with the ferroxyl indicator. In other words, as fast as the iron sweeps into solution the concentration of ferrous hydroxide grows, but the ferrous reaction appears in one place and the hydroxyl in another. It is now that the oxygen of the atmosphere dissolved in the solution takes up its work, the ferrous ions are oxidized to the insoluble ferric condition, which results in the precipitation of rust, and the action of hydrolysis proceeds. The formation of the insoluble ferroso-ferric carbonates and hydroxides, changing to the red ferric hydroxide known as rust, is familiar to everyone. Even the purest iron obtainable shows the electrolytic nodes in the ferroxyl indicator, which proves that the equilibrium is so delicate that it is affected by the physical condition of the surfaces as well as by the chemical constitution of the metal. In a recent publication[b] the writer has discussed this subject at length. It was observed that in making ferroxyl mounts an indentation, scratch, or wound of any kind on the surface of the steel invariably became positive to its surrounding area and thus formed a center of corrosion. If this had occurred only upon milled specimens which carried a coat of scale or blue oxide, the explanation would be simple, but the fact is, if a freshly polished steel mirror receives a cut before immersion, the marked place comes out in blue, and corrosion takes place more rapidly. It has been known for a long time that indentations or injuries on the water surfaces of boilers become centers of corrosion and pitting. It has been shown by Walker and others that the polarity is affected by stresses and strains on the surface, and it would seem, in view of all the facts, that this condition is determined by a number of widely different causes, which may be classified as electrical, chemical, and mechanical. It seems probable that the surfaces of steel which are subject to the condensation moisture of the atmosphere are always in a state of electrical strain and polarity. The ferroxyl tests show that in a large majority of cases the positive spots once formed remain positive, and so corrosion proceeds steadily to the formation of destructive pitholes.

Those who have followed the above explanation will understand the statement of the writer that the rusting of iron is primarily due to attack, not by oxygen, but by hydrogen in its ionized condition.

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[a] Wied. Ann., 1879, 7, 337.

[b] Trans. Am. Soc. for Testing Materials, 1908, 8, 244.

Nevertheless, the function of oxygen in carrying on the process of rusting, although a secondary one, is absolutely essential to its progress, for in the absence of oxygen rust is not formed. It is owing to these separate, though coordinated, functions of hydrogen and oxygen that the formation of rust can be inhibited by the addition to the water of certain oxidizing agents, as well as by certain reducing agents, a seeming paradox that no other theory attempts to explain. The strong reducing agent pyrogallol will inhibit rusting by interference with the function of the oxygen, while such conspicuous oxidizing agents as chromic acid and its soluble salts, by inducing a passive condition, interfere with the action of the hydrogen ions.

Let us now examine this theory in the light of some additional well-known facts. That the presence of acid is necessary for the continued corrosion of iron has been believed for a long time, and some authorities have even contended that carbonic acid is the sole cause of the rusting of iron. In the opinion of the writer this is true only inasmuch as carbonic acid is one of the factors in the hydrolysis which produces hydrogen ions. Iron does not rust in alkaline solutions which contain an excess of hydroxyl ions provided the alkalinity is high enough, but in dilute alkaline solutions rusting goes on in a dangerous fashion, the tendency to pit being accentuated. This action is incompatible with the theory that free acid is necessary to induce rusting, but is explained by the electro-chemical theory. Hydrogen and hydroxyl ions can exist in a solution at the same time to a considerable extent only when they are separated or held apart by potential differences, and therefore in the rusting of iron the concentration of hydroxyl ions, or, in other words, the alkalinity, must reach a certain value before rusting is entirely prohibited. This is because, owing to the very slight dissociation of water, an excess of hydroxyl ions would be incompatible with the presence of free hydrogen ions.

THE PASSIVITY OF IRON.

Chromic acid and solutions of its salts inhibit corrosion of iron and steel by inducing a passive condition of the surface. This passive condition is maintained not only while the metallic surface is in contact with the solution, but also for a certain length of time after the iron is clean and dry. The passive condition, however, gradually disappears. There is a vast literature[a] on the subject of passivity, and the three following theories have been advanced to account for it:[b] (1) A layer of oxide is formed on the surface; (2) oxygen is plated out on the surface, polarizing it to the condition of an oxygen electrode; (3) the solution tension of the iron is lowered by the change of the elementary iron to the condition of its higher valence, in which state it behaves as though it were a noble metal. The first theory is untenable, and each of the other two, though based on observed facts, offers incomprehensible difficulties. It is not, however, the intention of this paper to discuss these theories, but merely to present the subject in sufficient detail to permit the consideration of the practical bearing of the general theory on the important subject of the preservation of iron.

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[a] For the bibliography and discussion of the passive state see Byers, Jour. Am. Chem. Soc., 1908, **30**, 1718.

[b] Jones, H. C., Elements of Physical Chemistry, 1907, p. 441.

PRESERVATIVE COATINGS.

The various forms of iron and steel used in the arts and industries may be, for our present purpose, conveniently classified according to the methods in most general use for their preservation.

TABLE 1.—*Preservative coatings for various forms of iron and steel.*

Coatings.	Forms of iron and steel.
Zinc, tin, copper, lead	Roofing and sheathing of buildings; wire for telegraph, fencing, suspension cables, etc.; nails, screws, bolts, fittings, etc.; tanks, vessels, and utensils; chains for special purposes; pipes and tubes for special purposes.
Paints, varnishes, bitumens, cements, etc	Structural iron and steel; decorative ironwork, fire escapes, railings, etc.; locomotives and cars; smoke stacks, gas and oil tanks; pipes; machinery (in part); boats and ships; boilers (on the outside); trolley poles, masts, posts, mail boxes, etc.
Lacquers, varnishes, japans, and enamels	Small articles, hardware utensils, implements, tools, cans, etc.
Processed after manufacture to form a skin of oxide, phosphide, or some other compound	Structural steel, gun barrels, etc.
Usually unprotected	Machinery (in part); tools and farming implements (in part); chains; boilers and tanks (inside); railroad rails, plates, and bolts.

Taking up these forms of preservative coatings in the order indicated, we may inquire how successful the present methods of manufacture and preservation are, and whether or not the electro-chemical explanation of corrosion is likely to be of any practical value in indicating improvements in methods of protection.

It is unnecessary to discuss at length the dissatisfaction which is so widespread among consumers of modern steel and iron, who complain that it rusts and corrodes much more rapidly than older metals made largely by handworking in past times. The technical and scientific press is crowded with evidence of the truth of this contention. A prominent American railway engineer,[a] in considering the possible conservation of forest trees which would result from the substitution of steel for hard-wood railroad ties, has pointed out that unless more rust-resistant metal is manufactured such a course could not be economically followed. He further points out that the rate at which both wood and iron decay leaves little to be said in favor of either material, though while reforestation is possible, iron ore is not capable of reproduction.

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[a] Railroad Age Gazette, 1908, 45, 24.

The preservation of iron is, of course, a problem which, in the ease of its solution, varies directly with the rust-resisting character of the metal to be preserved. For this reason a number of the progressive steel manufacturers in the United States, and presumably elsewhere, have been for some time past investigating methods of manufacturing rust-resistant steel. It is not the writer's intention at this time to go deeply into this phase of the problem, but it seems to be a fact that carefully made, open-hearth metal, in which the ordinary impurities are cut down to mere traces and in which the heat treatment has been carefully controlled, is much more resistant to corrosion than the ordinary types of metal with a comparatively high percentage of impurities. The writer is well aware that reducing the content of impurities can only be achieved at the expense of tensile strength and perhaps other characteristics of steel as well, but for certain purposes great strength can be sacrificed to longevity.

There is now being manufactured in several rolling mills in the United States an extremely pure metal which runs, according to analysis, about 99.95 per cent iron. The only impurities which this metal contains are sulphur (0.019), phosphorus (0.004), and carbon (0.026.) This pure iron is manufactured in basic, open-hearth, 50-ton converters, and is smelted from pure pig iron and carefully selected high-grade scrap. There is no mystery about its manufacture, everything depending from start to finish on the heat treatment which the metal receives.

In the opinion of the writer, resistance to corrosion is attained largely by a study of the proper heat treatment of the metal and the amount of working which it receives in both the hot and cold rolls. Up to the present time this pure iron manufactured by a steel process has been used for the preparation of sheet and plate only. It has been demonstrated, however, that wire can be made from it. The demand for this type of metal appears to be growing, and it has been largely used for the manufacture of road culverts. In the letter of transmittal accompanying the writer's bulletin on the corrosion of iron, it was stated that the development of a sheet-iron culvert, which shall be at the same time strong, light, and resistant to corrosion, would be of great assistance to road builders. The fact that this new metal has been largely specified by culvert manufacturers, even at a somewhat added cost, is the best evidence that it is meeting a long-felt want in this and allied industries. There would seem to be no reason why such a pure iron, if it receives the proper heat treatment

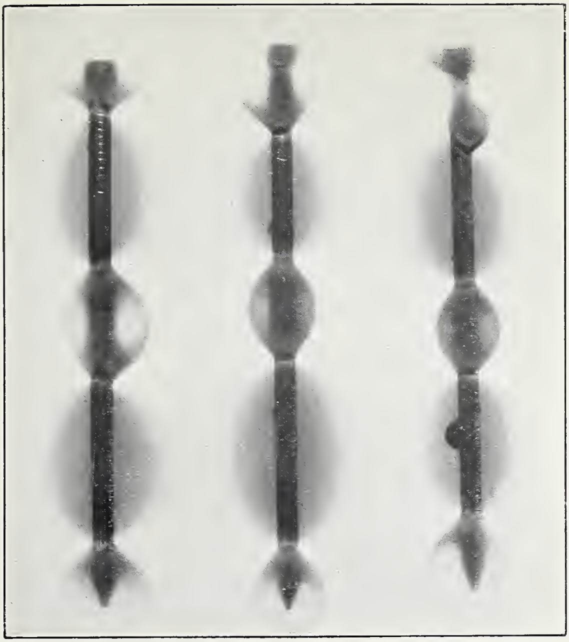


FIG. 1.—STEEL WIRE NAILS IN FERROXYL REAGENT.

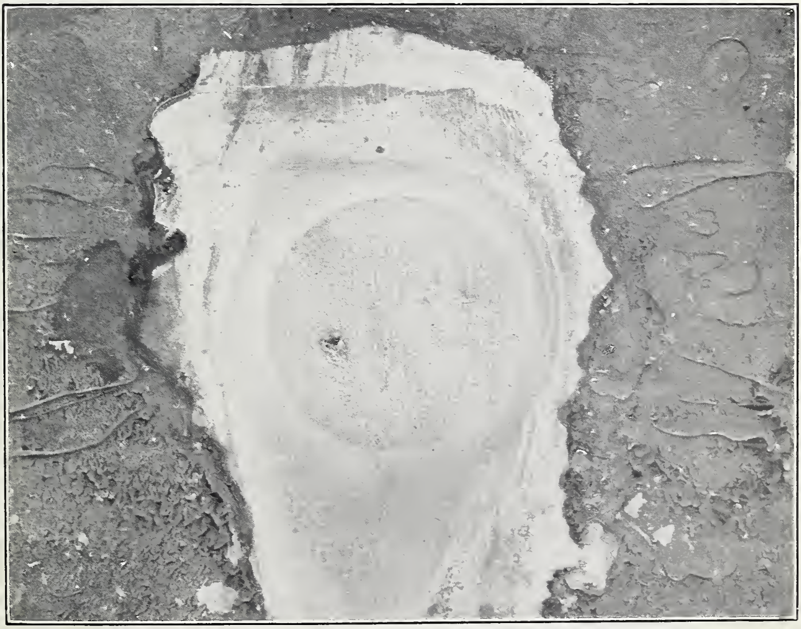


FIG. 2.—THE PROTECTION OF THE SURFACE OF STEEL IN THE IMMEDIATE NEIGHBORHOOD OF A BUTTON OF ZINC SOLDERED INTO THE STEEL.

and rolling, should not be found as resistant to corrosion as the pure iron made by the older processes.

Many metallurgists believe that the resistance to corrosion exhibited by some wrought irons is due to the slag and cinder inclusions which are always found in this type of metal. Microscopic examination does not, however, justify this conclusion, as the slag and cinder are found to lie in definite streaks or cleavage lines in the rolled iron. There is no evidence to show that the fibers of wrought iron are inclosed by cinder, as has been claimed by certain metallurgists. Impure wrought irons which have been prepared by what is known as the "busheling process" also contain the full amount of cinder inclusion, and yet are not at all resistant to corrosion. While it is undoubtedly true that the tendency of iron to rust is an elementary characteristic of the metal itself, there is nevertheless abundant evidence to show that very pure iron does not exhibit a tendency to dangerous pitting or localized corrosion to the same extent as more impure and carelessly manufactured steel.

Passing over the metallurgical and economic problems involved in the manufacture of rust-resistant steel, we will proceed to the discussion of the methods of preservation which depend upon covering iron with other metals.

PRESERVATION WITH ZINC.

As is well known, zinc is applied to the surface of iron by several different processes, which may be classified as the hot-dip method, misnamed galvanizing; the cold electro-plating method; and the vapor or dry-heat process, known as Sherardizing. Zinc is electropositive to iron, and when the two metals are in contact and wet with a corroding solution zinc will pass into solution and the iron will be protected. The sphere of influence of the zinc does not, however, extend far from the point of contact between the two metals, so that iron can begin to rust in places long before all the zinc is destroyed. This can easily be shown by soldering a button of zinc into the machined surface of a plate of steel and immersing the whole either in water or in the ferroxyl indicator. This action, which is very well illustrated in Plate I, fig. 2, shows how futile is the expedient of inserting zinc blocks in a boiler in the attempt to preserve the tubes. It is, of course, true that the more corrosive the action, the larger will be the sphere of influence of the protecting zinc and the faster the destruction of the zinc will proceed.

The function of the zinc coating in the ordinary galvanizing process is a double one; first, to cover the iron so that water and oxygen can not come in contact with it, and, second, to protect the iron at its own expense if pinholes or cracks in the zinc surface exist. Generally speaking, the more zinc that can be put on the more efficient the protection, but there are decided practical limits to the amount of zinc that can be carried by an iron surface. If the material must undergo bending in use, the heavier the coating the greater the danger of cracking. Thus, wire which is to be used in single strands, as, for instance, in telegraphy, can be "double galvanized" and unwiped, whereas wire which is to be woven into fences must carry a thinner coating and be wiped smooth. So-called "double galvanized" wire is not dipped twice into the bath of molten zinc, but merely carries about twice as much zinc by weight as ordinary wire. In the hot-dip galvanizing process it is customary in American practice to wet the iron with a soldering solution just before its immersion in the zinc bath. This soldering solution is generally an acid bath containing zinc chloride. The electro-chemical explanation of corrosion indicates that this is a dangerous practice for which metallurgists should find a substitute. That some of the electrolytes are included under the zinc coating can hardly be doubted. The system $Fe\text{-}FeCl_2\text{-}ZnCl_2\text{-}Zn$ is a galvanic couple which, however well it may justify the name of "galvanized iron," would certainly tend to hasten corrosion. The electro-chemical theory suggests the possibility of devising an inhibitive soldering solution as a substitute for the acid treatment. The writer, in cooperation with Mr. H. A. Gardner, has found that zinc oxide dissolved in an alkaline bath gives promise of a successful solution of this problem.

The cold galvanizing or electro-plating method of coating iron with zinc is open to the same criticism as the hot-dip method, inasmuch as the deposition is made from an acid bath with the inevitable inclusion of some of the electrolyte. The vapor deposition processes, such as that of Sherard, avoid this difficulty, but it is by no means certain that they do not introduce other dangerous factors. Although zinc is electro-positive to iron, certain alloys of zinc and iron are electro-negative. If these alloys are formed in the Sherardizing process, we defeat our main purpose in using zinc as a protective agent. At all events, the superior protective effect of zinc deposited by a vapor-coating process may be said, so far as the United States is concerned, to be still on trial. In the Sherardizing process the material to be coated is packed into air-tight iron drums together with zinc flue dust from the smelters. These drums are then heated for a certain length of time at a temperature below the melting point of zinc. The drums are also in some cases made to revolve while the heating is going on. By this means a very efficient coating of zinc is applied to the surface of the metal. For certain purposes this process may prove to be of value.

At the request of the writer a number of interesting tests have recently been undertaken for the purpose of ascertaining the relative resistance to corrosion of steel wires of varying chemical composition, and also to show the relative value of protective coatings.

With these ends in view 12 samples of galvanized steel wire were manufactured by a prominent steel and wire manufacturing company. They have been put up in the form of fence and exposed to the action of the weather. These 12 samples fall into 3 groups, which will be described in the order in which they were made.

The first sample was a wire of the following composition:

	Per cent.
Carbon	0.66
Manganese	.84
Sulphur	.028
Phosphorus	.016

This was made by the basic open-hearth method. The ingots were cast July 23, 1908, and were reheated and rolled into billets the same day. On the following day they were again heated to redness and rolled into $\frac{9}{16}$-inch rods. One billet was taken at random from the lot for the purpose of this test. The rod after cooling was cleaned by immersion in hot dilute sulphuric acid, then rinsed with water and dipped into milk of lime. After drying, the rod was drawn cold into 9-gauge wire. It was then galvanized by passing it through a furnace in which it was heated to dull redness, and then into a bath of hydrochloric acid containing zinc chloride, and lastly into a bath of molten zinc. This wire was not wiped; that is to say, it was allowed to retain as much zinc as would adhere to it. This sample was designated C–1, and it is purposed to test it against any of the low-carbon, low-manganese wires to be described later, and which may for this purpose be regarded as C–2.

The second group consists of six samples of "American" style, 8-strand, fabricated fencing, 45 inches high. The object of this group is to determine the effect, if any, of segregation of the impurities in the iron. Therefore the six samples were taken from the same heat. The steel was made at the same mill and on the same day as sample C–1, but by the Bessemer process, and was cast in six ingots. As these ingots were rolled, two billets were taken from the top of the first ingot, two billets from the middle of the first ingot, and two from the bottom of the first ingot. These three pairs of billets were marked, respectively, A–1, A–2, and A–3. Similarly, from the last ingot of the heat two billets were taken from the top, two from the middle, and two from the bottom. These three pairs of billets were marked, respectively, B–1, B–2, and B–3. These were all rolled hot into rods, and after cooling and cleaning, in the manner described above, were drawn into wire. The six samples were each divided into three portions. About half of each sample was drawn into 11-gauge wire, a quarter of each sample into 9-gauge wire, and the remaining quarter into 12-gauge wire. This was because these three sizes of wire are all used in making the standard "American" fence, the 9-gauge for the top and bottom horizontal strands, the 11-gauge for the intermediate strands, and the 12-gauge for the vertical connecting wires. Immediately after being drawn the wire was galvanized in the same manner as described for sample C–1, except that on emerging from the molten zinc it was wiped by a mechanical device which left just a thin, smooth, coating of zinc on the steel. A separate piece of fencing about 300 feet in length was then woven from each of the samples. These six pieces of fencing then represented the top, middle, and bottom of the first and last ingots of the heat, respectively. The samples were not analyzed separately, but an analysis of the heat as a whole showed percentages as follows:

	Per cent.
Carbon	0.09
Manganese	.55
Sulphur	.045
Phosphorus	.092

The third group consists of five samples of basic open-hearth steel showing increasing amounts of manganese from 0.07 per cent to 0.37 per cent. These were all made in one furnace, between August 18 and August 22, 1908. The process of manufacture throughout was identical with that employed on the sample designated C–1, except that the wire was wiped during the process of galvanizing. These five samples were all drawn to 11-gauge and are designated by their "heat numbers," which are given here together with the analyses.

TABLE 2.—*Analyses of five samples forming the third group.*

Heat numbers.	Carbon.	Manganese.	Sulphur.	Phosphorus.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
8118	0.05	0.12	0.014	0.014
8119	.04	.07	.014	.014
8120	.05	.16	.010	.010
8121	.05	.24	.018	.018
8122	.06	.37	.013	.016

All the wire described above was shipped to Pittsburg, Pa., and between September 23 and September 26, 1908, was put up in the grounds of the Carnegie Technical Schools. The site assigned for this purpose is a level strip of ground at the bottom of a deep and narrow natural depression to the north of the quadrangle of school buildings. Because of this location none of the wire will be exposed

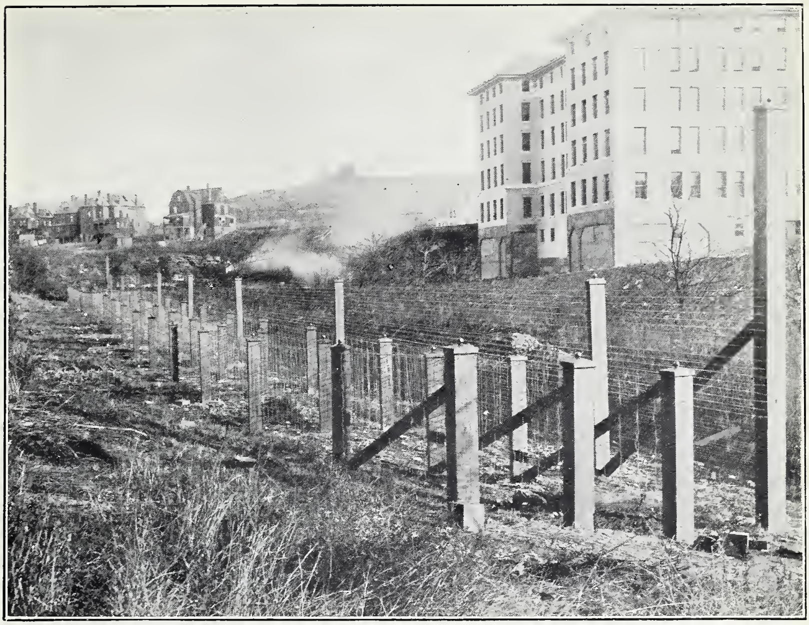


FIG. 1.—TEST FENCES AT PITTSBURG, PA.

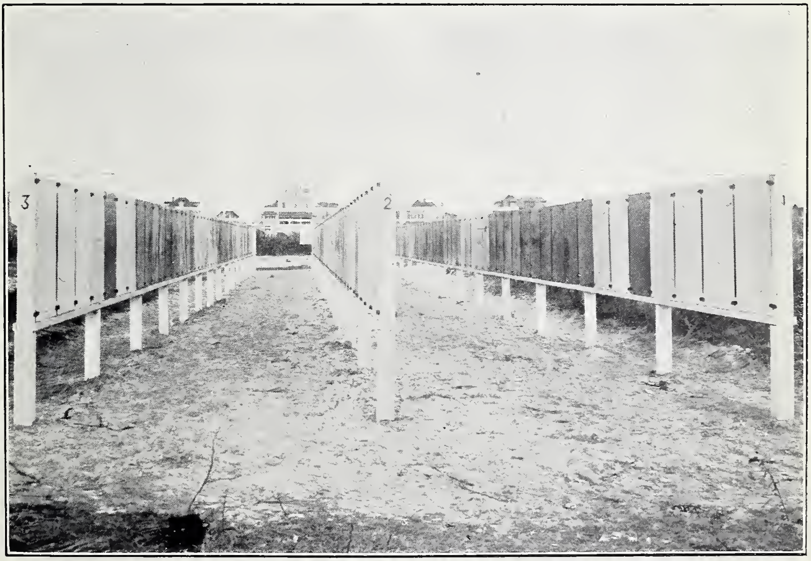
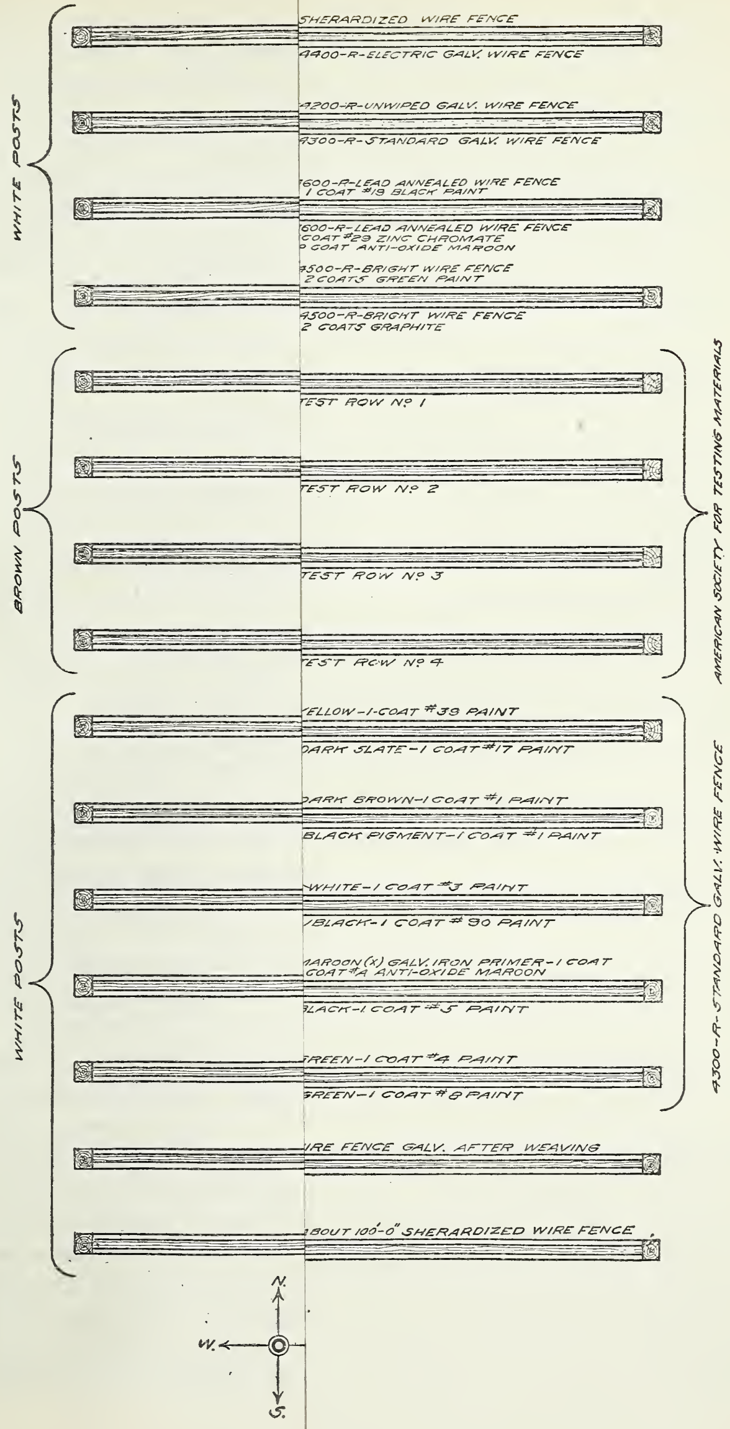
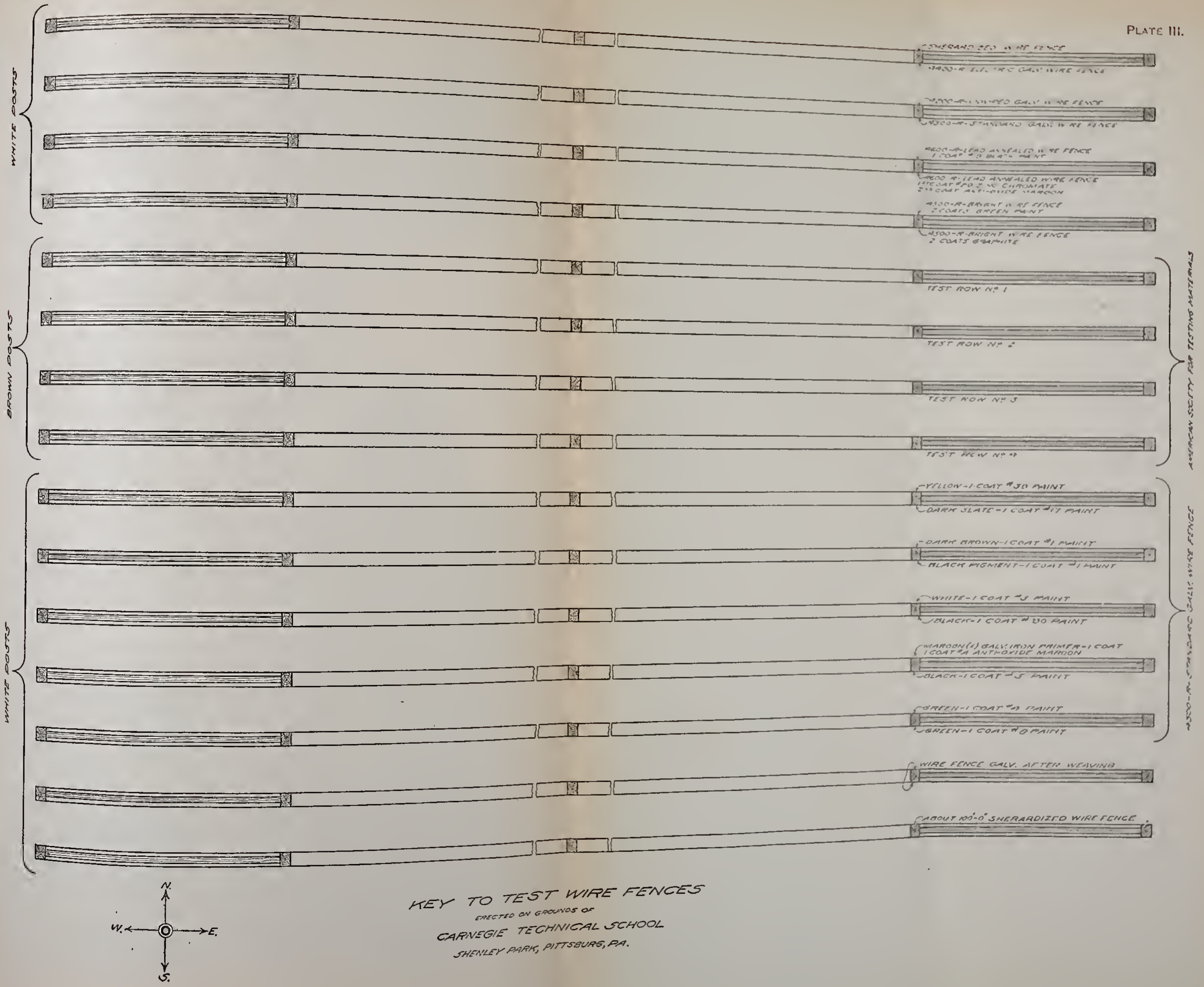


FIG. 2.—TEST FENCES AT ATLANTIC CITY N. J.


WHITE POSTS
SHERARDIZED WIRE FENCE
4400-R-ELECTRIC GALV. WIRE FENCE
4200-R-UNWIPED GALV. WIRE FENCE
4300-R-STANDARD GALV. WIRE FENCE
600-R-LEAD ANNEALED WIRE FENCE
1 COAT #19 BLACK PAINT
600-R-LEAD ANNEALED WIRE FENCE
COAT #29 ZINC CHROMATE
COAT ANTI-OXIDE MAROON
4500-R-BRIGHT WIRE FENCE
2 COATS GREEN PAINT
4500-R-BRIGHT WIRE FENCE
2 COATS GRAPHITE
BROWN POSTS
AMERICAN SOCIETY FOR TESTING MATERIALS
TEST ROW No 1
TEST ROW No 2
TEST ROW No 3
TEST ROW No 4
WHITE POSTS
4300-R-STANDARD GALV. WIRE FENCE
ELLOW-1-COAT #39 PAINT
ARK SLATE-1 COAT #17 PAINT
ARK BROWN-1 COAT #1 PAINT
BLACK PIGMENT-1 COAT #1 PAINT
WHITE-1 COAT #3 PAINT
BLACK-1 COAT # 30 PAINT
AROON (X) GALV. IRON PRIMER-1 COAT
COAT #A ANTI-OXIDE MAROON
LACK-1 COAT #5 PAINT
REEN-1 COAT #4 PAINT
REEN-1 COAT #8 PAINT
IRE FENCE GALV. AFTER WEAVING
BOUT 100'-0" SHERARDIZED WIRE FENCE
N.
W.
S.


WHITE POSTS
BROWN POSTS
WHITE POSTS
SHERARDIZED WIRE FENCE
4500-R-ELECTRIC GALV. WIRE FENCE
4500-R-ANNEALED GALV. WIRE FENCE
4500-R-STANDARD GALV. WIRE FENCE
4500-R-LEAD ANNEALED WIRE FENCE
1 COAT #9 BLACK PAINT
4500-R-LEAD ANNEALED WIRE FENCE
1ST COAT #PD ZINC CHROMATE
2ND COAT ANTI-OXIDE MAROON
4500-R-BRIGHT WIRE FENCE
2 COATS GREEN PAINT
4500-R-BRIGHT WIRE FENCE
2 COATS GRAPHITE
TEST ROW Nº 1
TEST ROW Nº 2
TEST ROW Nº 3
TEST ROW Nº 4
AMERICAN SOCIETY FOR TESTING MATERIALS
YELLOW-1 COAT #30 PAINT
DARK SLATE-1 COAT #17 PAINT
DARK BROWN-1 COAT #1 PAINT
BLACK PIGMENT-1 COAT #1 PAINT
WHITE-1 COAT #5 PAINT
BLACK-1 COAT #30 PAINT
MAROON(4) GALV. IRON PRIMER-1 COAT
1 COAT #A ANTIOXIDE MAROON
BLACK-1 COAT #5 PAINT
GREEN-1 COAT #A PAINT
GREEN-1 COAT #8 PAINT
4500-R-STANDARD GALV. WIRE FENCE
WIRE FENCE GALV. AFTER WEAVING
ABOUT 100'-0" SHERARDIZED WIRE FENCE
N.
W.
E.
S.

KEY TO TEST WIRE FENCES
ERECTED ON GROUNDS OF
CARNEGIE TECHNICAL SCHOOL
SHENLEY PARK, PITTSBURG, PA.

to the action of wind, but all will show the effects of fog and dampness, which are very prevalent in this hollow.

The fence was put up in an extremely substantial manner. The posts are 2 by 8 inches, dressed oak; the corner posts are 8 by 8 inches, sunk 3 feet in the ground. The lower ends were painted with a mixture of benzine and asphalt, and the portion above ground with ordinary lead paint. The post holes were filled in with concrete.

There are four parallel lines of posts, as shown in Plate II, fig. 1, 3 feet apart and 275 feet long. These lines run almost due east and west. The one farthest north is called row No. 1, the next row No. 2, the next row No. 3, and the line farthest south row No. 4. The arrangement of the wire on these posts is as follows:

On the north side of row No. 1 is placed sample A–1.
On the south side of row No. 1 is placed sample A–2.
On the north side of row No. 2 is placed sample A–3.
On the south side of row No. 2 is placed sample B–1.
On the north side of row No. 3 is placed sample B–2.
On the south side of row No. 3 is placed sample B–3.
On the north side of row No. 4 at the top are 8 strands of 8119.
On the north side of row No. 4 at the middle are 8 strands of C–1.
On the north side of row No. 4 at the bottom are 8 strands of 8118.
On the south side of row No. 4 at the top are 8 strands of 8122.
On the south side of row No. 4 at the middle are 8 strands of 8121.
On the south side of row No. 4 at the bottom are 8 strands of 8120.

In the first three rows the posts are 15 feet apart and in the fourth row 30 feet apart. The wire was all stretched as tightly as possible to prevent sagging, and none of it is less than 1 foot above the ground.

In addition to the test fences described above, a number of panels have been mounted on either side of the original rows in order to test different methods of providing protective coatings for ordinary steel wire fences. One panel of American style woven-wire fence is galvanized by the cold or electro-plating process; another panel is made of double galvanized wire carrying about the same weight of zinc as is usually specified for telegraph wire. This galvanizing has been done by a special adaptation and variation of the ordinary hot-dip process, and has not been subjected to the acid bath before galvanizing. A third panel is coated with zinc by the Sherardizing process. A number of panels, in part galvanized by the ordinary hot-dip process, and in part ungalvanized, have been protected by painting with various protective paint coatings. The formulæ of these stimulative and inhibitive paints will be published in a special report. All of these extra panels have been mounted in exactly the same manner and side by side with the samples already described in the foregoing, and the tests should in the course of time yield results of great value. A key to the arrangement of all the panels is shown in Plate III.

PRESERVATION WITH TIN.

Roofing, cans, and tinned utensils of all kinds depend for their preservation on a metal coating which, though not electro-positive to iron, has such a low solution tension that it is known to be practically incorrodible. The difficulty with the use of this metal is that it seems impossible at the present time to apply it economically to the surface of steel so that pinholes shall not exist in the coating. Walker has shown this by an ingenious modification of the ferroxyl test.[a] A slightly acid solution of gelatin containing a small amount of potassium ferricyanide is flooded, while hot, upon the surface of the tin plate to be tested. The gelatin becomes hard upon cooling, and in a very short time every pinhole on the tin surface is marked by a spot of Turnbull's blue. After the number and location of the pinholes are known it becomes possible to study ways and means of eradicating them. Hot rolling and aging of the plates under pressure are among the solutions that have been suggested in the hope that the pinholes will disappear.

The passage of the food and drugs act of 1906 has had a curious and interesting bearing on the tin-plate industry in its application to canned goods. Artificial coloring matter is debarred by this law, and it was found that the natural color of preserved foodstuff is reduced and destroyed by the action of the tin. In the effort to meet this difficulty the can manufacturers sought to use less tin, and also to lacquer the tin plate, so as to prevent its contact with the fruit and vegetable juices. The formation of pinholes has gone on rapidly, however, so that it is not an unusual occurrence to find tinned preserves discharging their juices in tiny streams through small holes resulting from rapid corrosion. Walker's method of examination should prove of great value to manufacturers who have to deal with a problem of this kind.

PRESERVATION WITH COPPER, LEAD, AND OTHER METALS.

It is possible to make a copper-coated steel wire, but this makes an efficient and rust-resistant wire material only when the coating is thorough and homogeneous. Methods are being developed to extend the use of copper for this purpose, and it is also applied now to sheet and plate metal. If the cost is not a prohibitive factor there would seem to be no reason why steel protected in this way should not come into more general use. The expedient of dipping iron into copper chloride or sulphate baths to supply a preliminary coating of copper before covering with zinc has also been tried. Lead has to some extent been used for covering iron, and for certain purposes this material is useful. It is doubtful, however, whether the application of lead in this way will find a very wide field. Methods have also been tried of applying an alloy of lead and antimony, but the success of the processes is not at present assured. One company in America has already placed on the market steel sheet coated with this alloy, the success of which will be watched with interest. Alloys of tin, zinc, antimony, and aluminum are also being experimented with, and give some promise of success. In all these coatings, however, the use of acid corrosive soldering fluxes should be avoided.

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[a] Paper read at Baltimore meeting of the American Chemical Society, December, 1908.

PRESERVATION WITH PAINT COATINGS, VARNISHES, BITUMENS, LACQUERS, AND ENAMELS.

The application of paint coatings of various kinds constitutes the method which is most generally depended upon for the preservation of iron and steel. It is along this line that the electro-chemical theories of corrosion appear to find the most important and interesting application. Two years ago, in a paper presented to the American Society for Testing Materials,[a] the writer suggested that, owing to their inhibitive action against rusting, the slightly soluble chromates should be theoretically the best protectives for the first application to iron and steel surfaces. Subsequent investigations, however, showed very conclusively that if the chrome pigment had been precipitated in an acid medium, or for any other reason contained soluble impurities that tended to stimulate corrosion, the surface of the steel might still fare badly. The following account of some special experiments,[b] a report of which has previously been published, will probably be found interesting in this connection. Samples of bright steel wire were immersed in 100 c. c. of a very dilute one-thousandth normal solution of potassium bichromate in a series of shallow dishes. The wire test pieces were suspended in the solution, so that they did not come in direct contact with the glass surfaces of the dishes. This precaution should never be omitted in experiments of this kind, as owing to the adsorption of air by glass rusting is always stimulated at the point of contact between glass and iron. The first dish was left as a blank, the second received one drop equal to 1/20 c. c. of a dilute tenth-normal copper sulphate solution. The third dish received two drops of the solution, and so on, each dish getting an increased amount of copper sulphate until 25 dishes had been prepared.

Now it is apparent that we have in this system two contending forces at work. Iron has a higher solution tension than copper and therefore tends to pass into solution, the copper tending to plate out on the iron. Chromate ions, on the other hand, put the surface of iron in a condition in which it can not pass into solution. In the solution system iron-chromate-copper we have an equilibrium to be decided between two contending forces acting in opposite directions. It was interesting and instructive to note the results of this struggle which was known to be going on, although the actual conflict could not be watched. In the first dish, in which no copper was present, no corrosion took place; in the second also no action was visible. In the third, however, minute specks of iron rust appeared. These were larger and more frequent in the immediately succeeding dishes, the test pieces showing rust tuberculation with the well-known pitting effect. As the middle of the series of dishes was approached, both iron rust and precipitated copper began to appear side by side on the surface of the iron, and from there on in the series more and more copper separated, while less and less rust formed, until in the end dishes copper and iron were changing places evenly over the surface without apparent hindrance. These experiments and others of a similar nature were repeated many times with the same results, and there seems to be no escape from at least the following two conclusions, to which they obviously lead:

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[a] Trans. Am. Soc. for Testing Materials, 1907, 7, 213.

[b] Trans. Am. Soc. for Testing Materials, 1908, 8, 605.

(1) If the surface of iron is subjected to the action of two contending influences, one tending to stimulate corrosion and the other to inhibit it, the result will be a breaking down of the defensive action of the inhibitor at the weakest points, thus localizing the action and leading to pitting effects.

(2) While the concentration of an inhibitor may be strong enough to prevent the electrolytic exchange between atom and ion, it must be still stronger to prevent entirely the solution of iron and the subsequent oxidation which leads to the formation of rust spots.

If these principles are applied to the selection of inhibitive pigments it is apparent that many paints may contain substances well adapted for carrying on just such a struggle on the surface of iron. It seems to be the consensus of opinion among experts that an ideal vehicle for spreading pigments does not exist. Linseed oil, well adapted as it is in some respects, not only undergoes complex changes, but absorbs and carries water. Therefore, even when covered with the best paints the surface of iron is sooner or later subject to attack by the two factors which produce corrosion, viz., hydrogen ions and oxygen.

These considerations suggested that more study should be given to the action of the various pigments which are in general use for compounding paints to be used on steel. A modified form of a test first proposed by G. W. Thompson[a] was used in the laboratory investigation. A series of bottles, all of the same size, were arranged with two-hole stoppers so that a current of air could be drawn rapidly through the whole series. Glass tubes connected one bottle to the next in line on the principle of a train of wash bottles. About fifty typical samples of pigments were obtained in the open market for the test and after careful sampling were distributed among five independent chemists for investigation. The method of testing followed by each investigator was as follows:

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[a] Trans. Am. Soc. for Testing Materials, 1907, 7, 493.

The train of test bottles having been set up as described, into each bottle were inserted two carefully weighed samples of sheet steel, each containing 4 square inches of surface area. Approximately 10 c. c. of each pigment was then added in turn to a bottle of the train with 100 c. c. of distilled water, and when all was ready a rapid current of air was drawn through the train. The duration of the test varied among the different workers, some allowing it to run for seven days, others for three weeks. Finally the test pieces were removed, scrubbed with a soft brush, dried, and weighed. For the purposes of this test the losses in weight were assumed to be the measure of corrosion.

TABLE 3.—*Results from tests to ascertain the value of pigments as rust inhibitors.*

TEST NO. 1 (H. A. GARDNER).

Name of pigment.	Loss in grams.
Zinc chromate	0.0050
Calcium chromate	.0088
Zinc and lead chromate	.0094
Zinc and barium chromate	.0153
Lithopone	.0160
Ultramarine blue	.0247
Zinc lead white	.0842
Chrome green (blue tone)	.0860
Umber	.1365
Prussian blue (inhibitive)	.1438
Zinc oxide	.1524
Willow charcoal	.1694
White lead (Dutch process)	.2040
White lead (quick process, basic carbonate)	.2120
Barium chromate	.2333
Asbestine	.2394
Carbonith (white)	.2655
Lemon chrome yellow	.2767
Venetian red	.2782
Sienna	.2826
Orange chrome yellow	.2876
Mineral black	.3050
Red lead	.3112
Sublimed blue lead	.3177
Chrome green (yellow tone)	.3265
Bone black	.3392
China clay	.3493

TABLE 3.—*Results from tests to ascertain the value of pigments, etc.*—Cont'd.

Name of pigment.	Loss in grams.
Indian red	0.3546
Keystone filler	.3560
Calcium carbonate (precipitated)	.3574
Prussian blue (neutral)	.3584
Prince's metallic brown	.3774
Calcium carbonate (whiting)	.3828
Bright red oxide	.3878
Orange mineral (French)	.3950
Calcium sulphate	.3966
Ocher	.4022
Medium chrome yellow	.4090
Sublimed lead (basic sulphate)	.4176
Litharge	.4325
American vermilion	.4328
Graphite No. 1	.4342
Orange mineral (American)	.4416
Barium sulphate (barytes)	.4454
Carbon black	.5003
Chinese blue (stimulative Prussian)	.5113
Precipitated barium sulphate (blanc fixe)	.5247
Graphite No. 2	.5262
Naples yellow	.6482
Lampblack	.7180
TEST NO. 2 (A. S. CUSHMAN).	
Barium chromate	.0101
Zinc and lead chromate	.0277
Zinc oxide	.0296
Zinc chromate	.0300
Zinc and barium chromate	.0468
Zinc lead white	.1712
Litharge	.1932
Calcium sulphate	.2143
Prussian blue (inhibitive)	.2267
Chrome green (blue tone)	.2269
Prince's metallic brown	.2620
Orange mineral (French)	.2724
Willow charcoal	.2795
White lead (Dutch process)	.2895
Ultramarine blue	.3185
Bone black	.3245
White lead (quick process; basic carbonate)	.3352
Sublimed blue lead	.3425
Red lead	.3555
Calcium carbonate (whiting)	.3620
Chrome green (yellow tone)	.3670
Indian red	.3739
Medium chrome yellow	.3767
Lithopone	.3791
Asbestine	.4025
Lemon chrome yellow	.4067

TABLE 3.—*Results from tests to ascertain the value of pigments, etc.*—Cont'd.

Name of pigment.	Loss in grams.
American vermilion	0.4147
Orange chrome yellow	.4203
Orange mineral (American)	.4336
Prussian blue (neutral)	.4463
Chinese blue (stimulative Prussian)	.4559
Keystone filler	.4651
Venetian red	.4756
China clay	.4770
Naples yellow	.4800
Calcium carbonate (precipitated)	.4910
Sienna	.5432
Graphite No. 1	.5437
Sublimed lead (basic sulphate)	.5856
Barium sulphate (barytes)	.5883
Umber	.5961
Graphite No. 2	.6337
Carbon black	.6955
Bright red oxide	.7896
Mineral black	.8018
Ocher	.8408
Precipitated barium sulphate (blanc fixe)	.8806
Lampblack	1.3098

TEST NO. 3 (P. H. WALKER).

Zinc and barium chromate	.0034
Zinc chromate	.0094
Ultramarine blue	.0137
Zinc and lead chromate	.0153
Litharge	.0309
Barium chromate	.0429
Prussian blue (inhibitive)	.0448
Zinc lead white	.0515
Chrome green (blue tone)	.0548
Bone black	.0921
White lead (basic sulphate)	.0982
Sublimed blue lead	.1001
Zinc oxide	.1002
Prussian blue (neutral)	.1218
China clay	.1248
Lithopone	.1274
White lead (quick process; basic carbonate)	.1288
Medium chrome yellow	.1319
Chrome green (yellow tone)	.1348
Lemon chrome yellow	.1365
Keystone filler	.1366
Calcium carbonate (whiting)	.1384
Willow charcoal	.1439
Red lead	.1495
Orange mineral (French)	.1495
Umber	.1498
Naples yellow	.1512

TABLE 3.—*Results from tests to ascertain the value of pigments, etc.*—Cont'd.

Name of pigment.	Loss in grams.
Orange chrome yellow	0.1700
Orange mineral (American)	.1719
Asbestine	.1748
Calcium sulphate	.1759
White lead (Dutch process)	.1781
Calcium carbonate (precipitated)	.1828
Venetian red	.1955
Prince's metallic brown	.1983
Mineral black	.2017
Chinese blue (stimulative Prussian)	.2055
Ocher	.2119
Barium sulphate (barytes)	.2547
Graphite No. 1	.2606
American vermilion	.2612
Indian red	.2617
Lamp black	.2838
Bright red oxide	.2920
Sienna	.2949
Precipitated barium sulphate (blanc fixe)	.3132
Graphite No. 2	.3723
Carbon black	.4069
TEST NO. 4 (L. G. CARMICK).	
Zinc oxide	.0085
Zinc and lead chromate	.0085
Barium chromate	.0094
Zinc chromate	.0130
Zinc and barium chromate	.0140
Zinc lead white	.0856
Prussian blue (inhibitive)	.1130
White lead (Dutch process)	.1150
Chrome green (blue tone)	.1240
Willow charcoal	.1362
Prince's metallic brown	.1408
Bone black	.1413
Chrome green (yellow tone)	.1453
Orange mineral (French)	.1467
Litharge	.1584
Calcium sulphate	.1597
Red lead	.1717
Medium chrome yellow	.1763
Lithopone	.1792
White lead (quick process; basic carbonate)	.1848
Ultramarine blue	.1865
American vermilion	.1877
Indian red	.1905
Orange chrome yellow	.1907
Lemon chrome yellow	.1972
Orange mineral (American)	.2065
Chinese blue (stimulative Prussian)	.2195
Asbestine	.2240

TABLE 3.—*Results from tests to ascertain the value of pigments, etc.*—Cont'd.

Name of pigment.	Loss in grams.
Naples yellow	0.2347
Sublimed blue lead	.2365
White lead (basic sulphate)	.2372
Venetian red	.2375
Calcium carbonate (whiting)	.2380
Prussian blue (neutral)	.2415
China clay	.2445
Calcium carbonate (precipitated)	.2625
Graphite No. 2	.2789
Sienna	.3085
Graphite No. 1	.3173
Keystone filler	.3349
Mineral black	.3529
Bright red oxide	.3707
Carbon black	.3751
Umber	.3817
Barium sulphate (barytes)	.3841
Ocher	.4315
Precipitated barium sulphate (blanc fixe)	.5085
Lampblack	.7096

TEST NO. 5 (W. H. WALKER).

Zinc and barium chromate	.0351
Zinc chromate	.0396
Zinc lead white	.0456
Ultramarine blue	.0496
Zinc oxide	.0504
Zinc and lead chromate	.0620
Calcium carbonate (whiting)	.1208
Keystone filler	.1481
Venetian red	.1564
Barium chromate	.1932
Prince's metallic brown	.1974
Willow charcoal	.2110
Orange chrome yellow	.2150
Calcium sulphate	.2174
White lead (quick process; basic carbonate)	.2274
Medium chrome yellow	.2288
Orange mineral (American)	.2315
Chrome green (blue tone)	.2346
Umber	.2403
Orange mineral (French)	.2526
Calcium carbonate (precipitated)	.2616
Prussian blue (inhibitive)	.2671
White lead (Dutch process)	.2743
White lead (basic sulphate)	.3116
China clay	.3212
Asbestine	.3405
Chrome green (yellow tone)	.3521
Mineral black	.3553
American vermilion	.3587

TABLE 3.—*Results from tests to ascertain the value of pigments, etc.*—Cont'd.

Name of pigment.	Loss in grams.
Naples yellow	0.3846
Prussian blue (neutral)	.4173
Indian red	.4334
Bone black	.4401
Bright red oxide	.4429
Sienna	.4462
Precipitated barium sulphate (blanc fixe)	.5064
Graphite No. 2	.5095
Chinese blue (stimulative Prussian)	.5202
Barium sulphate (barytes)	.5636
Red lead	.5707
Carbon black	.5716
Lamp black	.6257
Carbonith (white)	.7152
Graphite No. 1	.7165

The results obtained by the five separate investigators are shown in Table 3. Test No. 1 was made by Mr. H. A. Gardner, Philadelphia, Pa.; test No. 2, by the writer in the laboratories of the Office of Public Roads; test No. 3, by Mr. P. H. Walker, Bureau of Chemistry; test No. 4, by Mr. L. G. Carmick, also in the Office of Public Roads; and test No. 5, by Dr. W. H. Walker, Massachusetts Institute of Technology.

From these results a tentative classification of the pigments was made under the headings: (1) Inhibitors, (2) indeterminates, (3) stimulators. No one of the fifty pigments tested was, however, admitted to any class except in the cases in which a majority of the investigators reached the same result. The classification reached by this method is shown in Table 4.

TABLE 4.—*Classification of pigments.*

Inhibitors.	Indeterminates.	Stimulators.
Zinc lead chromate.	White lead (quick process; basic carbonate).	Lampblack.
Zinc oxide.	Sublimed lead (basic sulphate).	Precipitated barium sulphate (blanc fixe).
Zinc chromate.	Sublimed blue lead.	Ocher.
Zinc and barium chromate.	Lithopone.	Bright red oxide.
Zinc lead white.	Orange mineral (American).	Carbon black.
Prussian blue (inhibitive).	Red lead.	Graphite No. 2.
Chrome green (blue tone).	Litharge.	Barium sulphate (barytes).
White lead (Dutch process).	Venetian red.	Graphite No. 1.
Ultramarine blue.	Prince's metallic brown.	Chinese blue (stimulative Prussian).
Willow charcoal.	Calcium carbonate (whiting).	
	Calcium carbonate (precipitated).	
	Calcium sulphate.	
	China clay.	
	Asbestine.	
	American vermilion.	
	Medium chrome yellow.	

It should be carefully noted at this point that well-known pigments, such, for instance, as red lead and litharge, which fell into the indeterminate class, are not thereby impliedly condemned, nor, indeed, are the results of the work given out as a proof of any contention whatsoever. The results should be considered only as a step in a comprehensive investigation, and as preliminary to further studies which are about to be described. It is justly claimed by men of practical experience that laboratory tests on a small scale should not be used to draw sweeping deductions which may not accord with the results of experience, and which may, indeed, even introduce into practice methods of doubtful value. While admitting the general truth of this argument, it may be claimed that careful and systematic experiments based upon a sound theory are sure to yield fruitful results. It is interesting to note that in so far as they are comparable these results show a striking agreement with the English experiments carried out in 1899 and described by Smith.[a]

In order to extend these investigations into the field of practice, 600 large plates, representing three kinds of steel, were mounted in wooden frames facing the seashore near Atlantic City, N. J. These plates were painted under the strictest test conditions with the single paints as well as with formulæ made up from the three tentative classes of pigments. The following organizations cooperated to a greater or less extent in this work: United States Department of Agriculture, American Society for Testing Materials, Paint Manufacturers' Association of the United States, Masterpainters' Association of Pennsylvania, Carnegie Steel Company, and American Rolling Mill Company. The test plates, which were completed and exposed to the weather and salt air in September, 1908, are being watched with interest, and it is hoped and believed that they will yield results of much value to the metallurgist, the engineer, and all persons interested in paint technology. In view of the fact that these tests have been described in detail in a preliminary report[b] of the scientific section of the Paint Manufacturers' Association, it is unnecessary to describe them further at this time.

If we apply the autoelectrolytic theory of the cause of corrosion to the subject of protection by paint films, it follows that in general pigments that are *good conductors of electricity should never be applied directly to the surface of iron or steel.* A moment's thought will serve to show why this must be true, provided the theory is true. It has been shown that the surface of iron or steel, when brought into contact with an electrolyte, tends to throw itself into positive and negative nodes, so that spreading out a good conductor on the surface in the presence of moisture and oxygen will tend to stimulate corrosion at the positive nodes, with the well-known pitting or tuberculation effect always noted when steel rusts under atmospheric conditions. Whether the conductor is of the first class, such as powdered aluminum or graphite, or of the second class, such as a soluble inorganic salt, makes little difference, as either will stimulate the solution pressure of iron at the positive points. In the light of this theory let us consider the probable effect of using three forms of carbon pigments—lampblack, graphite, and willow charcoal—in contact with moisture on the surface of steel.

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[a] Jour. Soc. Chem. Ind., 1899, 18, 1093.

[b] Preliminary report on steel test fences, Paint Manufacturers' Association, Philadelphia, Pa.

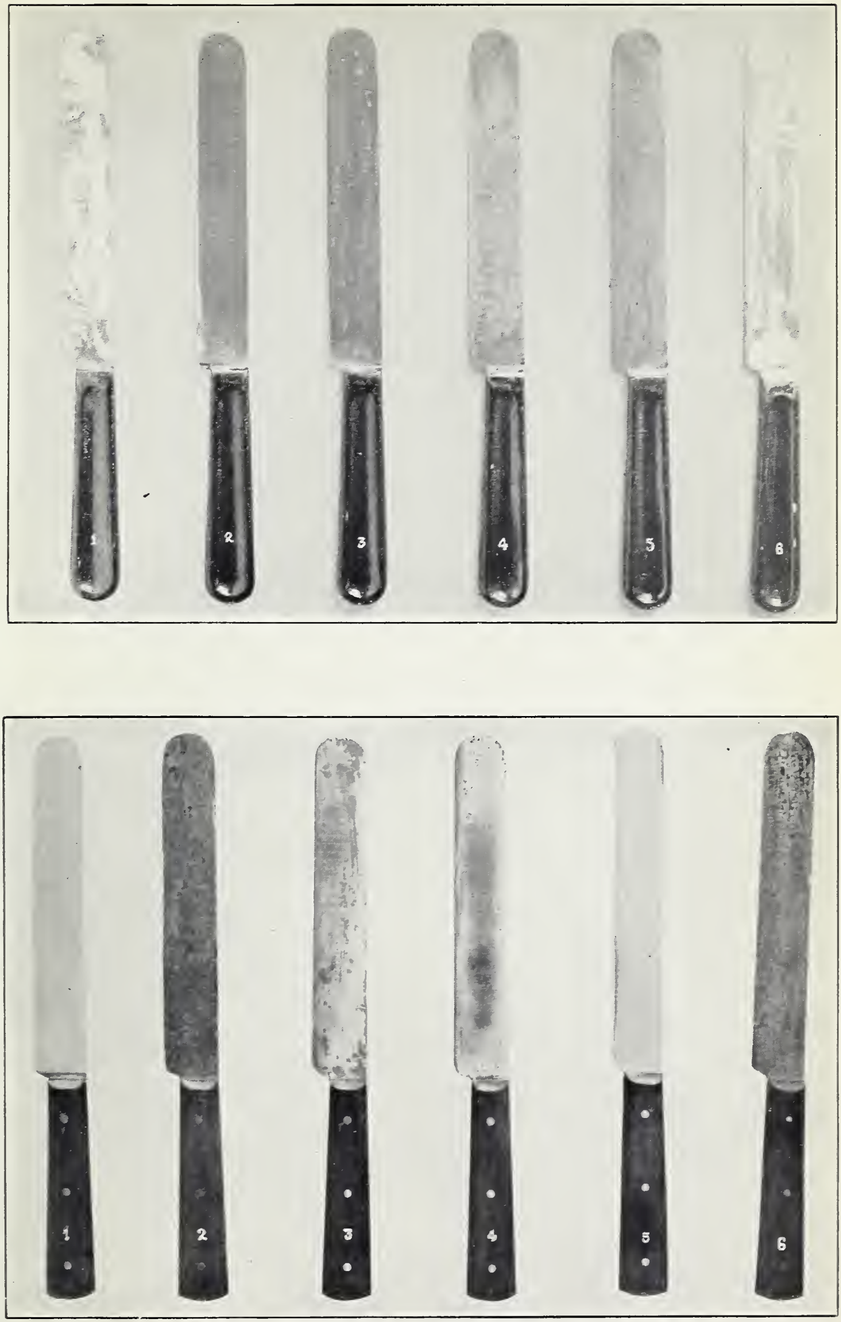
Of these three pigments in the dry state, lampblack and graphite have a high conductivity, while willow charcoal is almost an insulator. In order to get an approximate measurement of their relative conductivities in the moist state, 10 c. c. of each of the three pigments was shaken up with 100 c. c. of very pure distilled water and the conductivity of the resulting slimes quickly measured in a special type of conductivity cell. The results obtained are shown in Table 5.

TABLE 5.—*Results of conductivity test of three pigments.*

Name of pigment.	Comparative resistance.	Comparative conductivity.
Lampblack	0.44	2.28
Graphite	1.10	.91
Charcoal	1.83	.54

It will be noted that in the results shown in Table 3, in the results obtained by three out of the five experimenters, the corrosive action of the three pigments falls in this same order. In every case the lampblack and graphite appeared high up in the "stimulator" class, while willow charcoal just qualified as an "inhibitor." If we inquire why willow charcoal exhibits a slight conductivity when damp, and yet is to some extent an inhibitor of rust, the answer is that all charcoals contain a slight amount of potash. It is admitted that the conductivity test could not be generally used in order to determine the corrosive effect of a given pigment, for everything must necessarily depend upon whether the material which passes into solution, and thereby increases the conductivity of the water, is stimulative or inhibitive in action. Thus, alkaline electrolytes as well as chrome compounds inhibit, while acid and neutral salts generally stimulate corrosion.

Nevertheless, the test of the three carbon pigments as given above is of value, inasmuch as it indicates that good conductors should not be used in the first application to iron and steel, unless the conducting material is inhibitive in its nature. In order to determine whether or not this indication would be borne out by actual paint tests, the following experiments were made: The three carbon pigments were



KNIVES USED IN ACCELERATION TEST.

ground in the usual manner with a good grade of raw linseed oil and a small amount of a good drier. These three paints were then thinned slightly with pure turpentine and painted in neighboring stripes on samples of very pure steel which had been machined and polished to a smooth surface. After the paint films were set the plates were laid flat and covered with damp blotting paper, under which they were kept for three weeks. At the end of this time the paint films were removed with the usual paint solvents and the surface carefully examined. The steel was deeply etched under the graphite, less so under the lampblack, and to a much less extent under the willow charcoal. During the course of this work, it occurred to the writer that if an acceleration test could be devised, to distinguish between inhibitive and stimulative pigments, by a simple and easy method, and without the necessity of referring the matter to a chemist, it would be of great value to both manufacturers and consumers. To this end the following test has been devised:

The pigments to be tested are rubbed up with sufficient water to make thick water-color paints and are then flowed or brushed upon the clean blades of steel table knives. After the coatings are dry the knives are laid on a wet blotter and covered with a sheet of wet blotting paper. At the end of forty-eight hours the surfaces are cleaned off with running water and a stiff brush. This acceleration test has been found to give results which agree with the oil film tests. The following table indicates the pigments that were used. The first six knives tested are shown in the upper part of Plate IV, and the last six in the lower part of the same plate.

TABLE 6.—*List of pigments used in acceleration test.*

No. of knife.	Pigment.	No. of knife.	Pigment.
1	Barium chromate.	1	Zinc and barium chromate.
2	Zinc lead chromate.	2	Chrome green (blue tone).
3	Zinc chromate.	3	White lead (Dutch process).
4	Lampblack.	4	Red lead.
5	Graphite No. 2.	5	Litharge.
6	Willow charcoal.	6	Ferro-ferric oxide.

These film-corrosion tests usually agree with the results obtained in the Thompson test, and in the few cases in which differences have been noted reasonable explanations are forthcoming. For instance, in the cases of some of the very heavy pigments, such as the leads, barium compounds, etc., it was impossible in the air-bubbling test to keep them in suspension, with the consequence that the steel was in contact merely with the supernatant liquid. The damp-film test is much easier and more rational than the bubbling test and will probably come into very general use.

The fact has been brought out that some pigments which have been supposed to be excellent for protecting steel should in reality never be used, at least for the prime or contact coat, and that some pigments which theoretically should inhibit corrosion in reality appear to stimulate it. This is very well shown in the chrome pigments, some of which appear to be inhibitors and some stimulators, depending upon the technology of their preparation and whether or not they contain stimulating impurities. This is clearly shown in Plate IV, in which it may be seen that some of the chrome pigments have produced deep etching, while others have acted as inhibitors.

As a result of a large number of these film tests, the following conclusions appear to be justified and are therefore advanced at this time:

(1) The name of type of pigment does not necessarily guarantee its inhibitive value.

(2) Pigments which are good conductors of the first class should not be applied as the prime or contact coating to iron and steel.

(3) If pigments contain material which is even in the slightest degree soluble in water, they should not be used unless their inhibitive effect on corrosion·has first been established by test.

The use of varnishes and bitumens on the surface of iron recommends itself on account of the well-known insulating properties of these materials. The author has succeeded in making an efficient inhibitive lacquer by dissolving chromic acid crystals little by little in a filtered alcoholic solution of shellac. Many cases are on record in which pure refined coal tar has given a good account of itself as a protective coating, and undoubtedly many of the proprietary bituminous paints and compounds are well adapted for application to iron. The reason that such materials are not in general use appears to be that they change their nature to some extent as they age, and are apt to become brittle or to produce the effect known as "alligatoring" under the action of sunlight and the atmosphere.

PROTECTION WITH CEMENTS.

The question whether steel embedded in concrete is protected from corrosion is of the highest importance. If steel reinforcements rust away it bodes ill for the future of many modern structures, owing to the impossibility of making inspections and repairs before the danger point is reached. The records of discussions before a number of engineering and scientific bodies show that there is conflicting evidence and opinion in regard to this subject. There can be no doubt that the reaction of unleached cement concrete is strongly alkaline, owing to the separation of free lime at the time of set. If this alkaline reaction is maintained, steel embedded in the concrete should remain uncorroded. If, however, as is sometimes the case, percolating waters find their way through the concrete the free lime will eventually be removed and dangerous rusting take place. Nails and other objects of steel will remain bright when immersed in lime water, and the writer has found that the addition of about 5 per cent of quicklime to soggy sour clays and soils will have a very decidedly protective effect on steel embedded in them. It is curious that this simple expedient has not been resorted to in order to prolong the life of steel pipe lines trenched in soggy clay soils where there is but little movement of soil and subsoil water. Cement paints and grouts have also been used to protect steel, but have not come into general use, owing to the difficulty in making them adhere without cracking and peeling off.

PROCESSING AFTER MANUFACTURE TO FORM A COATING OF OXIDE, PHOSPHIDE, SILICIDE, ETC.

A number of metallurgical processes, such as the Bower-Barf[a] and others, have been from time to time proposed and exploited for producing an oxidized surface on steel. As far as the writer is aware none of these have as yet been found practically successful, except for certain special purposes.

The Coslet process is said to consist in immersing the iron in a hot phosphorized solution containing an iron compound. The surface, it is claimed, is converted into a ferroso-ferric phosphate which is to some extent resistant to corrosion. Jouvé[b] has called attention to the fact that iron containing 20 per cent of silicon is not attacked by acids, and therefore such material theoretically should not be subject to rust. The writer has confirmed this deduction by experiment, and found that iron containing 10 per cent of silicon is almost incorrodible. Unfortunately such a metal is not easily workable and has peculiar properties. Since silicon is much like carbon, chemically speaking, it would seem as if it might be worked into the surface of steel by modifications of some of the processes used for case-hardening with carbon. If anyone should discover a method for applying a skin of high silicon metal on worked steel it would be a contribution of the highest value to the art of metallurgy and to the world at large.

UNPROTECTED STEEL.

A very large proportion of the iron and steel which is in use can not, from the very nature of the service to which it is put, be protected from corrosion. In this class belong rails, heavy chains, implements, boiler tubes, etc. The only hope of meeting this particular phase of the problem consists in the improvement of metallurgical processes, to the end that perfectly homogeneous metal as free as possible from segregation may be manufactured. It is well known that some of the alloyed steels, such as nickel steel, are very highly resistant to corrosion. The same thing is probably true of the chrome and chrome-vanadium steels. Unfortunately, such materials are too costly to be used on a large scale, although there is always the possibility that some alloy or combination will be discovered which will be at the same time comparatively inexpensive and incorrodible. It appears to be the opinion of a number of metallurgists with whom the writer has corresponded that carefully made open-hearth steels in which the ordinary impurities have been worked down to a minimum, with especial care in the heat treatment, are much more resistant to corrosion than the ordinary run of merchant steel. One well-known example of this type of steel which is on the market, and which has already been referred to in a previous paragraph, has the following analysis:

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[a] Wood, M. P., Rustless coatings, 1905, p. 166.

[b] Engineer, 1908, **106**, 397.

	Per cent.
Sulphur	0.019
Phosphorus	.004
Carbon	.026
Manganese	Trace.

This metal is therefore, practically speaking, 99.95 per cent pure. Although it is apparent that a metal of this character could not be used where a tensile strength in excess of 40,000 to 50,000 pounds per square inch is required, there are certain purposes for which it is well adapted, such, for instance, as roofing, sheathing, culverts, pipes, etc.

It has been found that this pure steel (or iron) resists the attack of acids to a high degree, and an acid test has been proposed to predetermine the resistance to corrosion of various types of metal. This proposal has, however, met with decided opposition from some metallurgists, who believe that the test is not a rational one. The need of a quick preliminary test for determining the resistance of steel and iron to corrosion is universally conceded. It is, however, in the opinion of the writer, extremely doubtful whether such a test is possible, in view of the fact that the autoelectrolysis which is slowly but continually at work in the natural process of rusting does not proceed in the same way in any short-time test. This criticism applies to all the various tests which have been proposed, such as immersion in corrosive liquids while air is being bubbled over the test pieces. A considerable amount of work has already been done in investigating this particular problem, and the results have already been in part published in the Transactions of the American Society for Testing Materials.[a] More work is being done and will soon be published. Up to the present time there would seem to be but one perfectly sure criterion, and that is the test of service.

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[a] Amer. Soc. for Testing Materials, 1908, 8, 231.

Electrolytic processes for protecting steel have also from time to time been proposed. As far as the writer is aware none of these have come into general use. It should of course be possible, by making the steel to be preserved the anode in an electric circuit against a scrap-iron cathode, to check, if not entirely stop, corrosion. It is, however, extremely doubtful if such a method could be made to work economically. Engineers are much disturbed by the damage to certain steel structures which is caused by electrolysis due to escaped currents from high potential circuits used in lighting, trolley lines, etc. This undoubtedly constitutes a very serious engineering problem, but it should be remembered that extraneous currents may be made to protect as well as to attack steel through electrolysis. Laboratory experiments indicate, however, that a considerable voltage is necessary to prevent a steel anode in an electrolytic circuit from suffering from corrosion. The action which has been described in this paper as autogenous electrolysis, or autoelectrolysis, may be truly said to be the underlying and active cause of all forms of corrosion.

CONCLUSION AND SUMMARY.

It has been shown in this paper that the preservation of iron and steel has a direct and important bearing on the conservation of the natural resources of the world. The controlling factors which influence the more or less rapid corrosion of iron and steel are discussed in the light of the electrolytic or autoelectrolytic theory of rust formation. The various methods which are in common use, or which have been proposed for the protection of iron and steel, are then considered with special reference to the results of recent investigations, and certain new principles are announced to serve as a guide in the selection of paints for the prime or contact coatings for steel. With regard to the protection of metal which, from the nature of its service, can not be treated with a protective coating, it is pointed out that this is purely a metallurgical problem looking to the manufacture of a perfectly homogeneous and carefully worked material.

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